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Design of an Adiabatic Demagnetization Cryostat for Ultrasonic Studies.

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DESIGN OF AN ADIABATIC DEMAGNETIZATION CRYOSTAT FOR ULTRASONIC ATTENUATION STUDIES

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DESIGN OF AN ADIABATIC DEMAGNETIZATION CRYOSTAT FOR ULTRASONIC ATTENUATION STUDIES

by

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ABSTRACT

A cryostat for low temperature research was designed with minimal changes to an existing He^3 refrigerator. The new design provides for isothermal magnetization and subsequent adiabatic demagnetization of a paramagnetic salt to reach temperatures as low as $0.06^{o}K$. The cryostat is to be used for ultrasonic attenuation studies of superconducting metals with low transition temperatures. The low temperature is necessary to obtain accurate comparison with the BCS theory, which predicts a constant zero degree superconducting energy gap for all superconductors of 3.50 kT.

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INTRODUCTION

Experimental results show a marked decrease in the attenuation of ultrasonic waves in superconductors below T_c, the superconducting transition temperature. Bardeen, Cooper, and Schrieffer (BCS) explained these results in 1957 as a decrease in the electron contribution to the ultrasonic attenuation. The BCS theory predicts that the ratio of the electron contribution to the attenuation in the superconducting state (α_s) to that in the normal state (α_n) is

$$\frac{\alpha_s}{\alpha_n} = \frac{2}{1 + \exp\left[\epsilon(T)/k_B T\right]} \qquad (1)$$

where $2\epsilon(T)$ is the temperature dependent energy gap. Furthermore, the theory predicts a zero degree energy gap of $3.50\ kT_c$.

The BCS prediction of a constant zero degree energy gap of $3.50\ kT_c$ for all superconductors, is based on the assumption that the phonon-electron interaction energy is constant and isotropic near the Fermi surface. However, Morse, Olsen, and Gavenda [1] showed that in tin there is considerable anisotropy of the energy gap ($3.2\ kT_c$ - $4.32\ kT_c$).

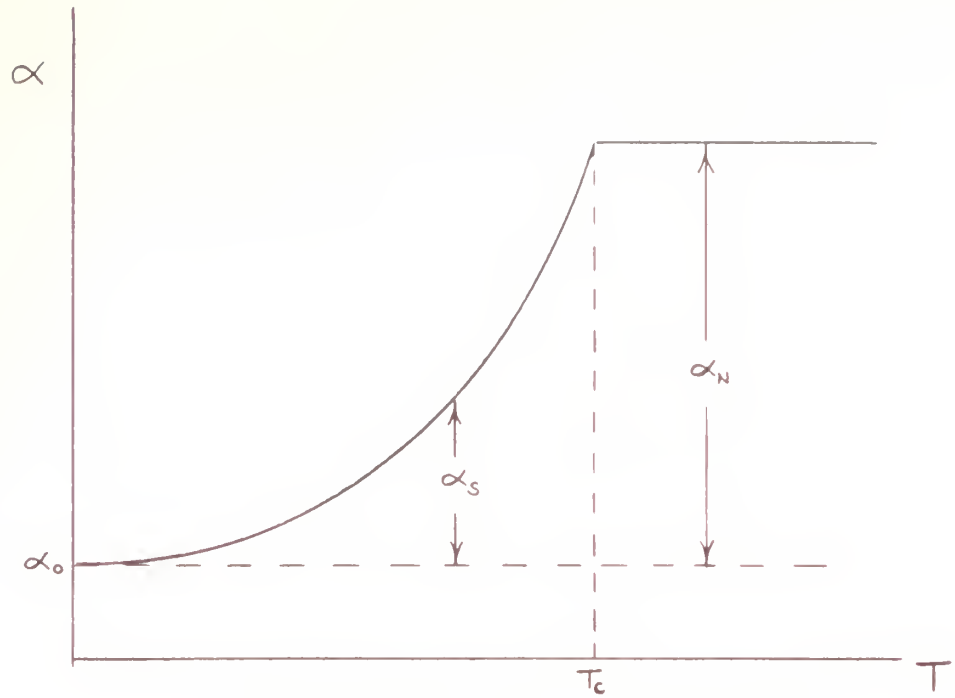
To conserve both momentum and energy in a phonon-electron interaction, the electron velocity component parallel to the direction of sound must be equal to the phonon velocity; i.e., the sound velocity. Therefore, only the electrons traveling essentially perpendicular to the direction of sound propogation can interact with the phonons, since the velocity of electrons near the Fermi surface is generally much greater than the sound velocity. The ultrasonic attenuation is an average over the narrow band perpendicular to the sound velocity in which these electrons lie. Hence, the energy gap measured will be the average for the electrons lying within this narrow band. Thus anisotropies

in the Fermi surface will lead to anisotropies in the energy gap. Temperatures below 0.1°K. will allow for dependable investigation of these anisotropies and the zero degree energy gap.

Figures 1 and 2 show, respectively, typical plots of experimental and theoretical investigation of ultrasonic attenuation in the superconducting state. The schematic experimental plot, Fig. 1, shows a residual attenuation, α_o, due to mechanisms other than electron-phonon interactions. This residual attenuation must be subtracted from the measured attenuation in order to analyze the results using equation (1). When the attenuation scales are normalized a plot similar to Fig. 2 is obtained. In Fig. 2, $\alpha^* = \alpha_s/\alpha_n$ and $t = T/T_c$. Thus, the ratio of attenuation due to conduction electrons alone in the superconducting and normal states is obtained for comparison of experimental results with the BCS theory.

The BCS theory makes the simple prediction that the variation of α_s/α_n with temperature should be $\alpha_s(t)/\alpha_n = 2f(\epsilon)$, where $f(\epsilon)$ is the Fermi function of $\epsilon(t)$, $2\epsilon(t)$ is the temperature-dependent energy gap, and t is the reduced temperature. Using this assumption, Muhlschlegel has tabulated values of the superconducting energy gap (in reduced coordinates) as a function of the reduced temperature. A plot of these values is shown in Figure 3, and it is seen that below about t=0.35 the slope of the plot has only a very slight deviation from zero.

A convenient method for determination of this temperature-dependent energy gap from attenuation measurements of α^* is to plot $Y = 1/(2/\alpha^* - 1) = [kT_c/\epsilon(t)]t$ versus t (Figure 4). Y is obtained by solving equation (1) for $\epsilon(T)/kT$, inverting the result, and substituting $T = T_c \cdot t$. The slope at t=0 for this plot gives the reciprocal of the zero degree energy gap in units of kT_c. According to Figure 3 this curve will be approximately



Absolute Attenuation vs Temperature

Figure 1

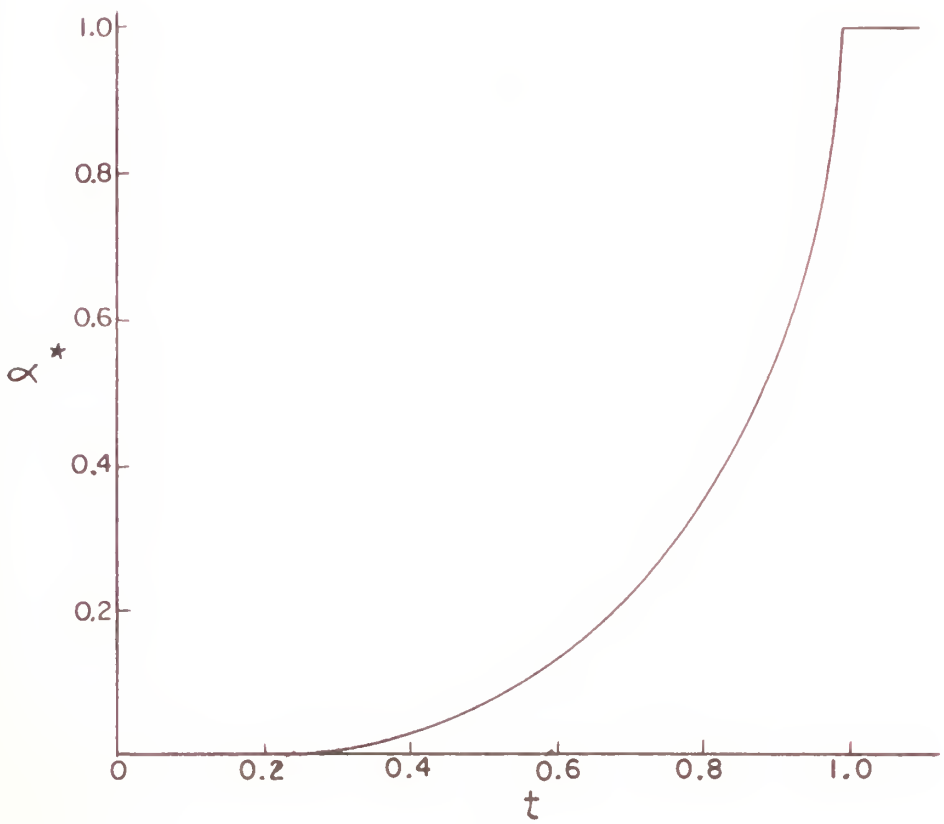
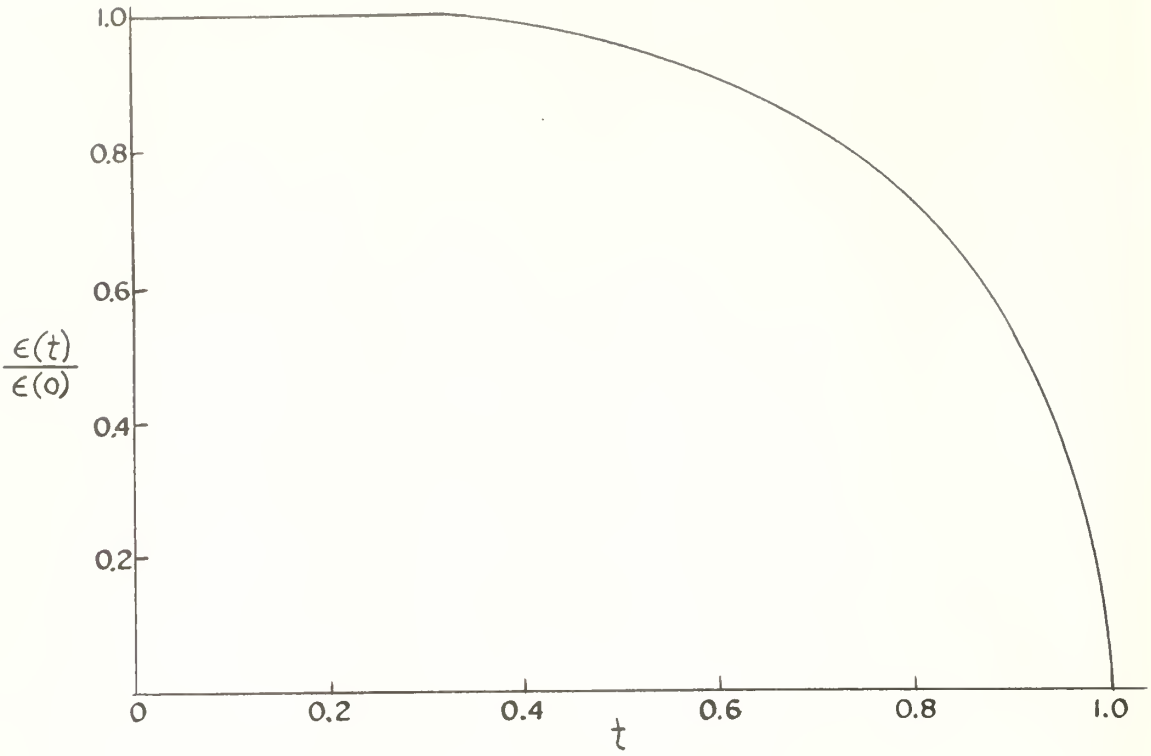


Figure 2

Reduced Attenuation vs Reduced Temperature



Variation of the Superconducting Energy Gap with Temperature

Figure 3

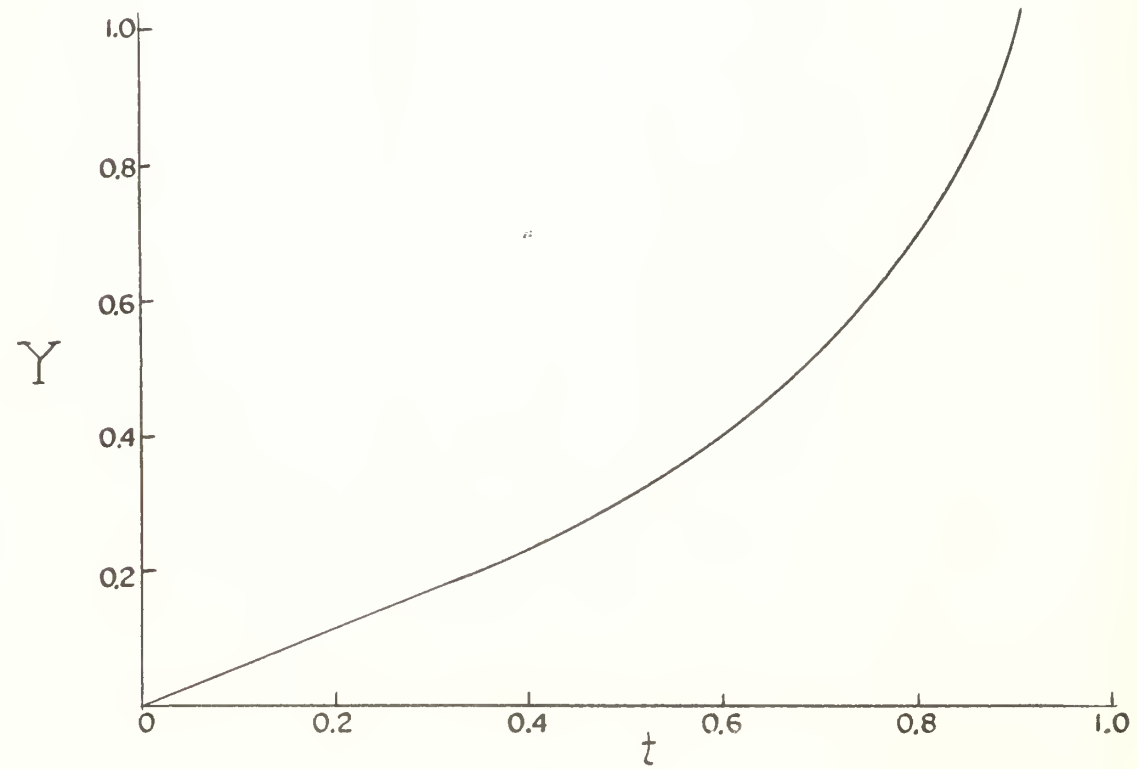


Figure 4

$Y = 1/\ln(2/\alpha^* - 1)$ vs Reduced Temperature

a straight line passing through the origin for t 0.35. For an accurate experimental determination of the zero degree energy gap, several values of Y below t = 0.35 must be obtained.

In experimental work by Goncz [2] on the ultrasonic attenuation in zinc a He^3 refrigerator was used, in which the lowest temperature reached was approximately $0.34^{\circ}K$. Since zinc has a transition temperature of about $0.87^{\circ}K$., the lowest value of t obtained was 0.39. Thus the linear region of the plot of Y vs. t could not be reached, and, therefore, the resulting extrapolation to obtain the zero degree energy gap was not too accurate. Had it been possible to obtain temperatures below $0.1^{\circ}K$. ($t<0.12$), many points in the linear region could have been obtained. The resulting value for the zero degree energy gap would be much more accurate.

By reaching temperatures below $0.1^{\circ}K$ accurate results of the zero degree energy gap for superconducting metals with Tc as low as $0.2^{\circ}K$. could be obtained. This would include the low Tc superconducting metals Cadmium (Tc = $0.56^{\circ}K$.), Molybdenum (1.0), Osmium (0.7), Ruthenium (0.49), Titanium (0.40), and Zirconium (0.75) as well as a large number of superconducting alloys.

Thus, in ultrasonic attenuation studies of superconducting metals it is essential to obtain as low a temperature as possible. The accuracy of the zero degree superconducting energy gap determination for metals with low transition temperatures can be greatly improved if temperatures below $0.1^{\circ}K$. can be reached. In addition, the greater accuracy would enable one to examine the differences between metals and anisotropies in a given metal. By obtaining experimental evidence for gap variations from one metal to another, as well as for gap anisotropies, one may be able to refine the details of the BCS calculations.

DISCUSSION

Theory and Design of the Adiabatic Demagnetization System

One of the experimental methods used in reaching temperatures below 0.35°K. is adiabatic demagnetization. A cryostat was designed which would be suitable for ultrasonic attenuation studies and capable of reaching temperatures of 0.06°K. Figure 5 is a schematic diagram of this cryostat.

A He^3 refrigerator is used to obtain a temperature of about 0.32°K. The paramagnetic salt is suspended below the He^3 bath by nylon tubing having a low thermal conductivity. The nylon tube, which contains the electrical leads for pulsing and for the resistance thermometers, extends through the salt to the sample holder. The magnet surrounds the salt and is suspended in the He^4 dewar.

The superconducting metal is loaded in the copper sample holder suspended below the paramagnetic salt and He^3 chamber. The vacuum jackets of the He^3 chamber and the He^4 dewar are flushed with dry nitrogen and left at about 100 microns dry nitrogen pressure. Then the nitrogen dewar is filled with liquid nitrogen and the whole system is cooled to approximately 77°K.

Then the He^3 and the He^4 vacuum jackets are pumped down to less than 5×10^{-6}mm. Hg. It is extremely important to have the He^3 jacket pressure less than this to prevent thermal conduction at the lowest temperatures. To prevent freezing out impurities in the He^3 Chamber, He^3 gas is circulated for about one-half hour with the nitrogen and He^4 traps filled. After the jackets are pumped out and the He^3 purified, the He^4 dewar is filled and the system cooled to 4.2°K.

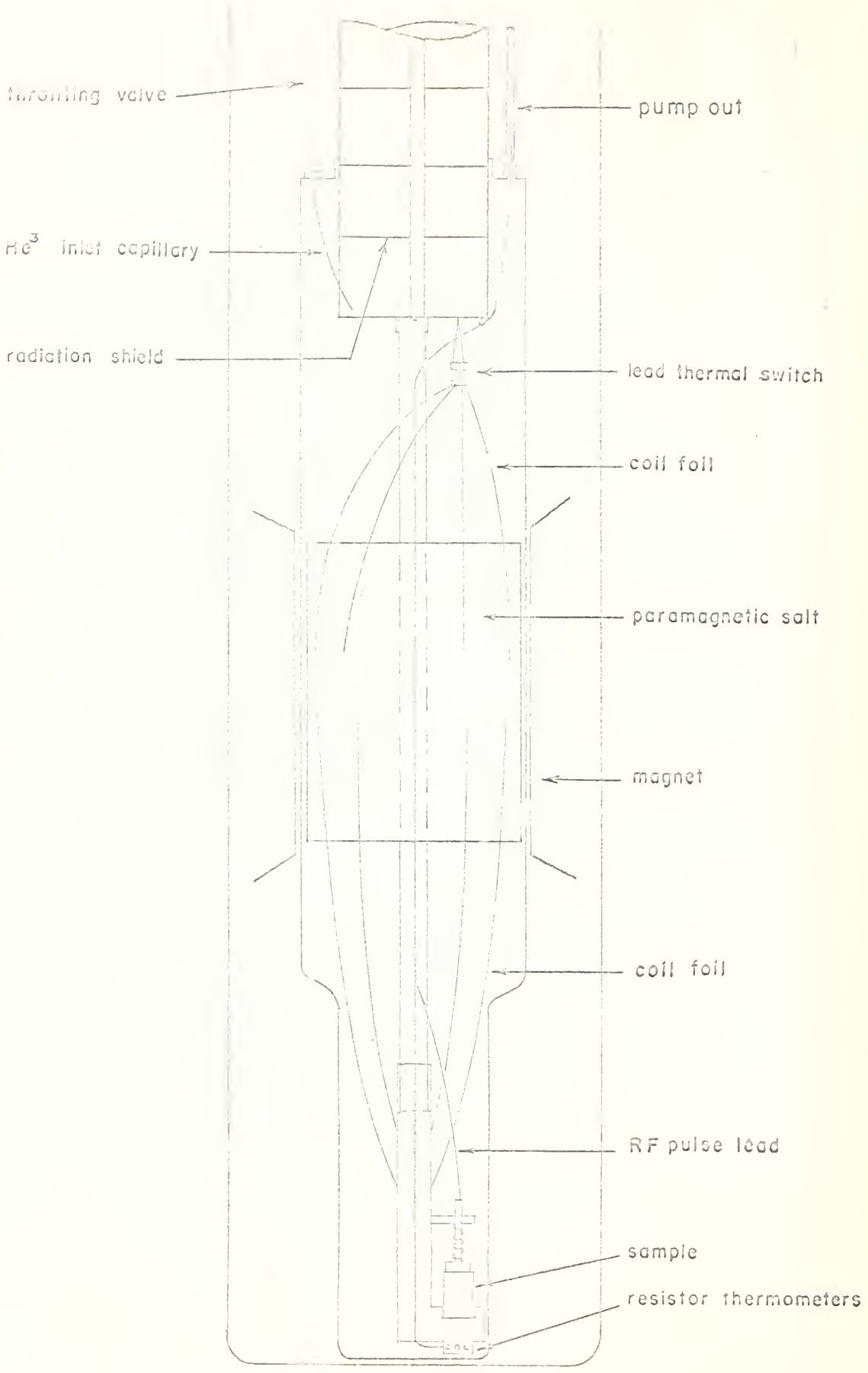


Figure 5. Schematic of Adiabatic Demagnetization Cryostat

When 4.2^{o}K. is reached, the He^{4} is pumped on and the He^{3} let into the He^{3} chamber. A minimum temperature of about 1.1^{o}K. can be reached in this manner, well below the temperature of condensation of He^{3} at one atmosphere (3.195^{o}K). When equilibrium is reached, the He^{3} is pumped on with the booster pump bringing the He^{3} chamber to a temperature of approximately 0.32^{o}K.

At this time the superconducting magnet is turned on for isothermal magnetization of the paramagnetic salt. A lead thermal switch is provided between the He^{3} chamber and the paramagnetic salt for stabilizing the temperature of the salt (and sample) in the presence of the large power input.

The thermal conductivity of the superconducting lead at the temperature well below its transition temperature (7.18^{o}K.) is much greater when in the normal state than in the superconducting state. Therefore, when the large magnetic field is applied, the lead is driven into the normal state and there is heat flow between the He^{3} chamber and the salt. The salt pill will come to equilibrium with the He^{3} bath, providing the temperature of 0.32^{o}K. for the isothermal magnetization. Then when the field is turned off, for the adiabatic demagnetization, the lead will return to the superconducting state thereby providing a minimum heat leak at low temperature of the salt.

Figure 6 shows what is happening thermodynamically during the isothermal magnetization, A to B (Change from H = 0 to H = 10 kilogauss at T_i) and then during the subsequent adiabatic demagnetization, B to C (change from H = 10 kilogauss at T_i to H = 0 at T_f). The magnetization process, by orienting the magnetic ions of the salt, decreases their disorder and thereby causes a reduction in entropy. The salt is

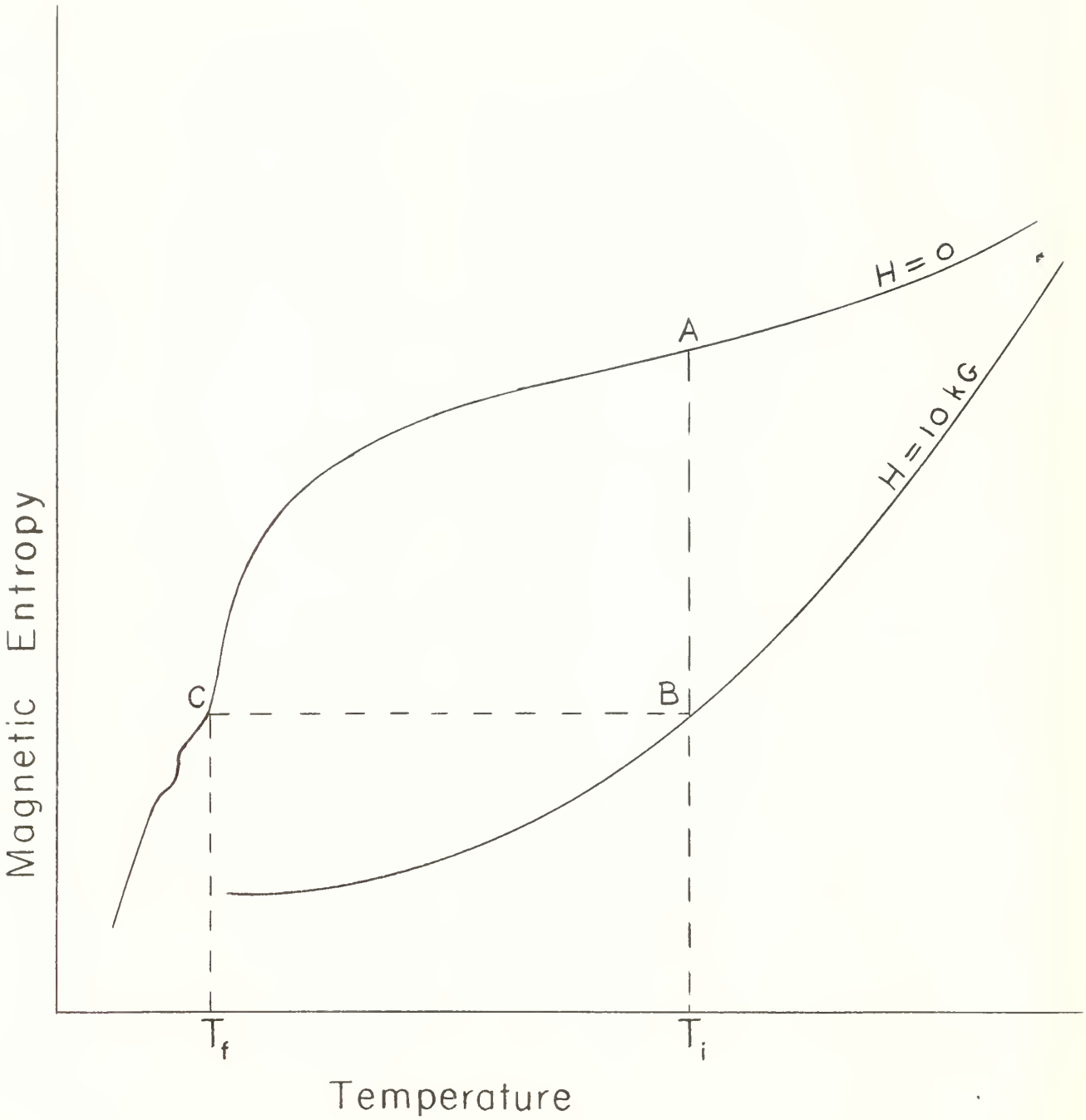


Figure 6. Magnetic Entropy vs Temperature

then thermally isolated and, therefore, when the magnetic field is reduced to zero the process follows the horizontal isentropic line and the temperature falls to T_f.

The entropy of the salt consists of two contributions: that form the magnetic (spin) system and that from the lattice. The two are assumed to be independent and, at temperatures below $1^{\circ}K.$, the lattice entropy is usually negligible compared to the magnetic entropy. In the paramagnetic salt, the spin-lattice relaxation time is short, that is, equality of spin and lattice temperatures is attained rapidly after a change in either. The field is applied in a time that is great compared with the time needed to transfer the heat of magnetization away to the He^3 bath (this time is normally of the order of minutes), thereby providing a nearly isothermal process.

Before any field is applied, the paramagnetic ions of the crystalline lattice in the salt have no preferred orientation. The interaction (dipole-dipole or exchange) is small enough so that the magnetic moment may take up any one of (2J + 1) directions, where J is the total angular momentum. The probability of the magnetic moment of an ion having a certain orientation follows the Boltzmann distribution, i.e., the factor $\exp(U/kT)$ where U is the energy difference between adjacent orientations (or energy levels). Since $U = g\beta H_{int}$ is much less than kT_o, any of the (2J + 1) orientations are equally probable. Here g is the Lande splitting factor and β is the Bohr magneton.

When the magnetic field is applied the paramagnetic ions will become at least partly oriented parallel to the field, i.e., the ions will go into the lower energy levels which correspond to dipoles pointing in the direction of the applied field. The splitting $g\beta H$ should be greater

than kT. Hence, the order of the salt is increased, the entropy decreased, and heat flows from the salt to the He^3 bath.

During the adiabatic demagnetization, the Boltzmann distribution of the ions over the energy levels remains the same, the magnetic moment is constant, and the energy differences between the levels remain proportional to the field, i.e., U returns to its original low value. At very low fields the interaction forces become of the same order as the external field strength, the differences between energy levels are no longer proportional to the field, and the ions are redistributed over the levels in such a way that the entropy is kept constant. Thus a final state is obtained where the entropy is lower than at the initial temperature in zero field and hence the temperature is lower.

FINAL TEMPERATURE OBTAINABLE

For a paramagnetic salt, the entropy, S, and the magnetic moment, M, are functions of H_E/T only (where H_E is the applied external field) down to a temperature θ. At the temperature θ, thermal energies are comparable to interaction energies. Above this temperature Curie's Law

$$M = \text{constant } H_E/T \tag{2}$$

is obeyed very closely, and ion-ion interaction is negligible. The magnetic specific heat is given by:

$$C_M = \left(\frac{\partial U}{\partial T}\right)M = T \left(\frac{\partial S}{\partial T}\right)M \tag{3}$$

Applying the second law of thermodynamics for an adiabatic process to equation (3) gives:

$$C_M dT = T \left(\partial H_E/\partial T\right)_M \, dM \tag{4}$$

Since a constant external field is applied, $(\partial H/\partial T)_M = (\partial M/\partial T)_M = dM/dT$. Therefore,

$$C_M = T \, d^2M/dT^2 = \text{constant}/T^2 \tag{5}$$

in the temperature region where Curie's Law is obeyed.

Figure 6 shows the thermodynamic process of attaining low temperatures by adiabatic demagnetization. At the final temperature the magnetic field is approximately equal to zero. The property of interest since the salt is to be used as a cooling medium is its specific heat. The specific heat in zero field, C_m, can be obtained from the relation:

$$C_m = T(\partial S/\partial T)_m$$

Thus from the slope of the H=0 curve in Fig. 6 the corresponding values of the specific heat can be obtained. A rise in entropy with temperature means a high specific heat. The steep rise in entropy occurs at the Curie point and, therefore, at this temperature there is a discontinuity in the specific heat. A plot of specific heat vs. temperature for ferric ammonium alum from experimental results of Kurti and Simon is shown in Fig. 7. The specific heat maximum occurs at approximately $0.04^{\circ}K$. Kurti and Simon further showed that below this Curie point for ferric ammcnium alum an adiabatic demagnetization produces heating, while above this point adiabatic demagnetization produces cooling.

Since the salt is used for cooling a sample of large heat capacity it is most efficient to use a salt whose specific heat maximum lies below the required working temperatures. It is also necessary to carry out the demagnetization reversibly so that the sample remains very close to the temperature of the salt during the demagnetization.

Ferric ammonium alum, $FeNH_4(SO_4)_2 \cdot 12H_2O$ was selected as the most suitable salt as it has the specific heat maximum occuring at the lowest temperature of the well-studied salts. Since the only isotope with nuclear spin, ^{57}Fe, is only 2.2% abundant, nuclear effects are negligible, and the specific heat is due almost entirely to Stark splitting and interactions between the ions. In addition, Curie's law is closely

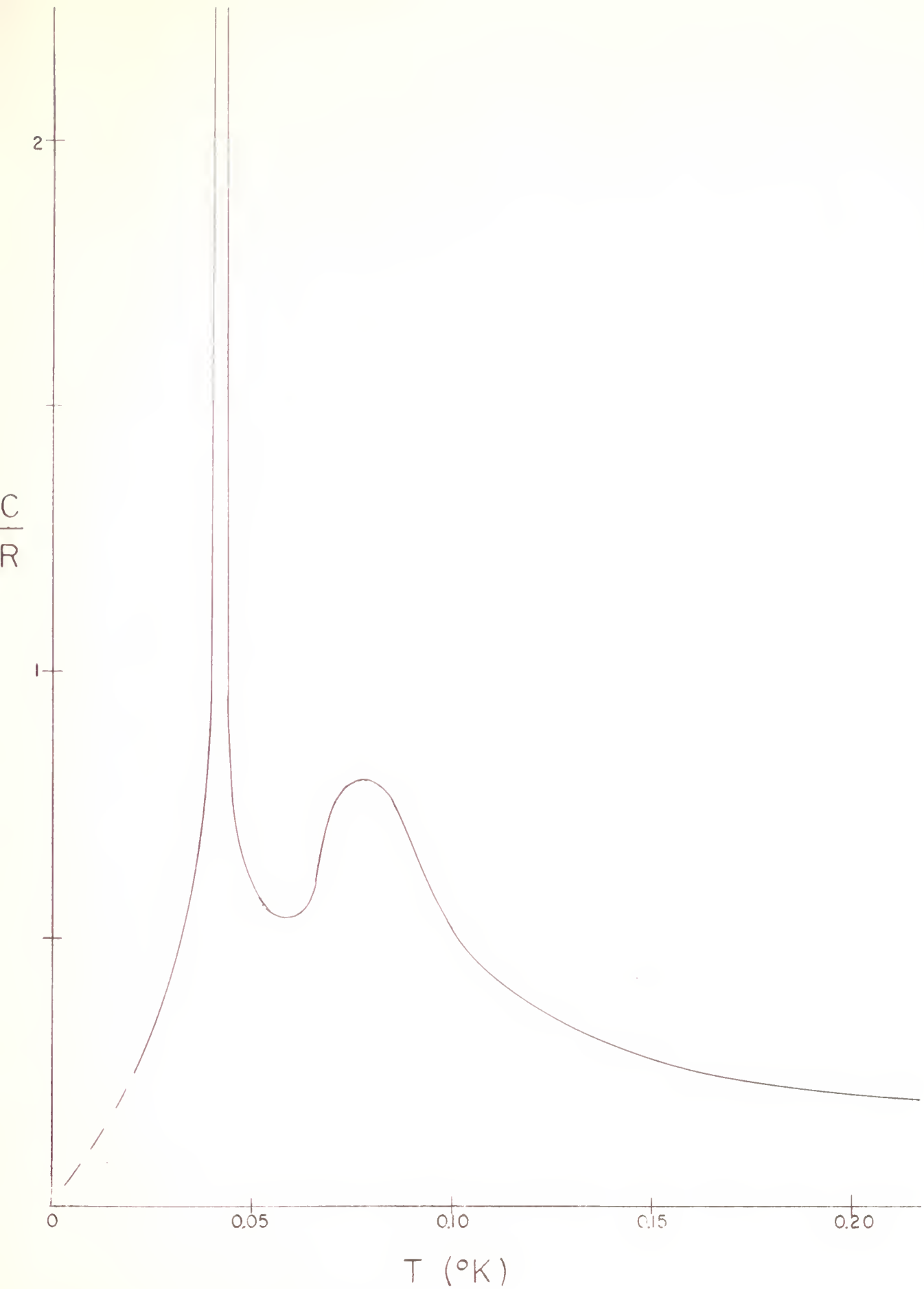


Figure 7. Specific Heat vs Temperature for Ferric Ammonium Alum

followed with a Curie constant of 4.37 $cm^3 deg/mole$. Measurements of specific heat by Kramer, Bijl, and Gorter [3], in the helium range give $C/R = 0.013/T^2$, in agreement with measurements by Benzie and Cooke [4]. These are also in close agreement with the demagnetization measurements of Kurti and Simon [5] of $C/R=0.014/T^2$. The limiting temperature, θ, was shown by Kurti and Simon to be approximately $0.06^{\circ}K$.

In addition, a large decrease of entropy can be obtained with the ferric salt during the isothermal magnetization. Since the ground state of the ferric ion is $^6S_{5/2}$ the number of possible configurations of the ion with respect to an external field is $2S + 1 = 6$. The total decrease of magnetic entropy is, therefore, $S_m = R \log (2S+1) = R \log 6$.

The properties of the salt may be altered by diluting it so as to increase the distance between magnetic ions, thereby decreasing the magnetic interaction and the splitting of lower energy levels. In this manner lower values of T may be obtained by increasing H. However, dilution also reduces the magnetic entropy resulting in a reduction of the total heat capacity/volume. Therefore, the capacity of the salt for cooling the sample would be reduced.

The thermal link for cooling the sample is provided by "coil-foil" connecting the salt with the sample. The "coil-foil" is constructed by winding approximately 500 turns/inch of No. 44 copper wire over .001 in. Mylar on a drum. The resultant coil is coated with a 1:1 mixture of General Electric 7031 varnish and toluene and then baked in an oven. The coil is cut parallel to its axis, and the Mylar is removed. The resultant foil is quite strong and provides a large contact area in the salt for ready flow of heat to the sample. In addition, eddy current heating is minimal.

ACKNOWLEDGEMENTS

I wish to express my sincere appreciation to Professor John R. Neighbours for his most helpful guidance and assistance. I would also like to thank Professor W. Reese and Mr. Lynwood Maye for many informative conversations.

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APPENDIX I

THERMOMETRY

Carbon resistance thermometers are used for temperature measurements as they are relatively insensitive to magnetic fields, are of sufficiently high resistance for the measuring power to cause negligible temperature rises, and the carbon has small heat capacity and short thermal relaxation time. One-half watt Speer Carbon Resistors, of nominal resistance 470 ohms, 10%, grade 1002 are used.

Temperatures are determined by measuring the resistance in a Wheatstone Bridge Circuit employing a lock-in amplifier to measure the bridge unbalance. The resistors are calibrated against the vapor pressure of He^4 down to 1.1°K. using the "1958 He^4 Scale of Temperature". Down to the lowest temperature obtainable, the resistors are calibrated against the magnetic susceptibility of cerium magnesium double nitrate (CMN), $Ce_3Mg_3(NO_3)_{12}\cdot 24\ H_2O$, using a Cryotonics Mutual Inductance Bridge.

APPENDIX II

ULTRASONIC PULSING EQUIPMENT

A schematic of the ultrasonic pulsing equipment to be used is shown in Figure 8. In this equipment, a trigger pulse initiates the horizontal sweep and simultaneously activates the pulsed oscillator. The pulsed oscillator then excites the quartz crystal bonded to the sample with an RF pulse of approximately two microseconds duration. The frequency of the pulse, tuned to the fundamental or odd harmonic of the quartz, is variable between 10 and 90 Mc/s. The quartz in turn generates an ultrasonic pulse in the sample and this pulse echoes between the two parallel faces of the sample. Each time the pulse returns to the quartz interface, a small voltage is generated which is received, amplified, and displayed (in proper time relation to the initial pulse) on the oscilloscope. The display shows a decaying train of pulses, each one of which has made one more round-trip through the sample than the preceding pulse. The change in height of each echo is a measure of the attenuation in the sample. Absolute attenuation is obtained by taking pictures of the scope and plotting echo heights vs. echo number on semi-log paper.

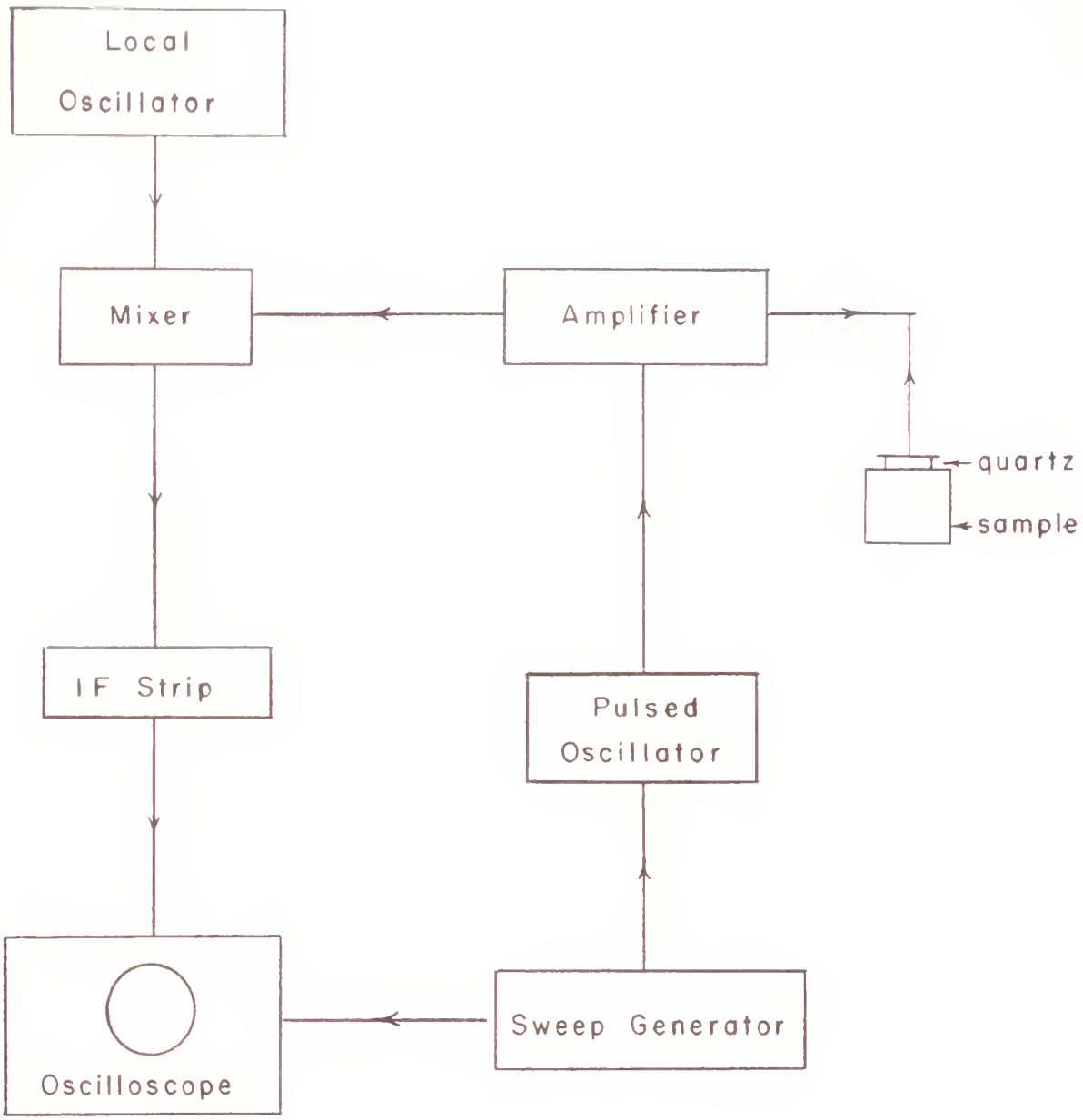

Figure 8. Ultrasonic Pulsing Equipment Schematic

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13. ABSTRACT

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